# Petroleum Analysis with Agilent PLgel Columns and Gel Permeation Chromatography

Application Note

Materials Testing and Research, Polymers


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## Introduction

Crude oil, or petroleum, is the main source of organic chemicals for industry. The major chemicals are derived from two constituents of oil, xylene, and naphtha. These raw materials are then broken down into more basic products, such as polyethylene, polypropylene, elastomers, asphalts, and liquid hydrocarbons. Characterization of such products is commonly achieved using gel permeation chromatography (GPC). This involves a liquid chromatographic separation from which a molecular weight distribution calculation can be made following calibration of the system with suitable polymer standards. The diversity of petroleum products demands a variety of Agilent PLgel GPC columns for optimized analysis.

## Analysis of Asphalt

Low molecular weight liquid hydrocarbons require high resolution of individual components. This is illustrated in Figure 1, where three linear hydrocarbons are resolved easily to base line in a reasonably short analysis time.

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## Conditions for Figure 1

| | |
|---|---|
| Columns | 2 × Agilent PLgel 5 µm 100Å, 7.5 × 300 mm (p/n PL1110-6520) |
| Eluent | 1,2,4-Trichlorobenzene |
| Flow rate | 1.0 mL/min |
| Temp | 100 °C |
| Detector | RI |
| System | Agilent PL-GPC 220 |

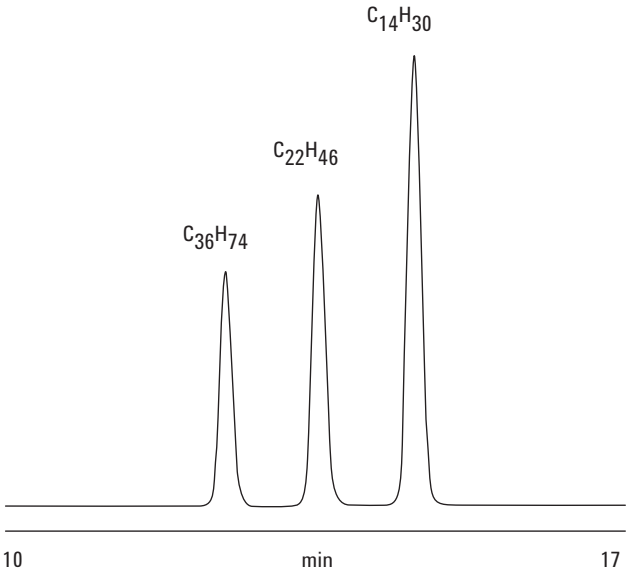


*Figure 1. Low molecular weight linear hydrocarbons resolved to base line using an Agilent PLgel 5 µm two-column set.*

The Agilent PLgel 5 µm 100Å columns have a GPC exclusion limit of 5,000 molecular weight (polystyrene equivalent), and efficiency is typically > 60,000 plates/m. Intermediate products can be analyzed using the Agilent PLgel 5 µm MIXED-D column, which has a linear molecular weight resolving range up to an exclusion limit of around 500,000 molecular weight. The 5-µm particle size maintains high column efficiency, therefore, fewer columns are required, and analysis time is relatively short.

Figure 2 shows the analysis of asphalt used in road surfacing. Subsequent information regarding the molecular weight distribution of such materials is invaluable in determining processibility and final properties.

## Conditions for Figure 2

| | |
|---|---|
| Columns | 2 × Agilent PLgel 5 µm MIXED-D, 7.5 × 300 mm (p/n PL1110-6504) |
| Eluent | THF |
| Flow rate | 1.0 mL/min |
| Temp | 50 °C |
| Detector | RI |
| System | Agilent PL-GPC 50 |

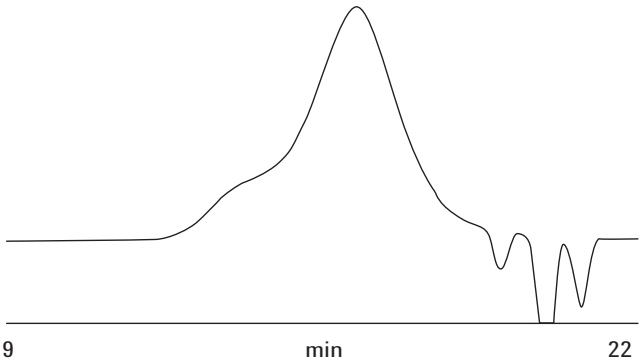


*Figure 2. Asphalt separation on an Agilent PLgel 5 µm MIXED-D two-column set.*

High molecular weight polyolefin polymers tend to exhibit very broad molecular weight distribution. In these applications, small particle size packings are not desirable since the incidence of polymer shear degradation is apparent. The Agilent PLgel 20 µm MIXED-A column is ideally suited, with a high exclusion limit (40,000,000 g/mol polystyrene equivalent). Its larger particle size, with subsequent lower efficiency, means that three or four columns are required in series. Figure 3 shows typical polyethylene and polypropylene analyses on Agilent PLgel 20 µm MIXED-A columns.

## Conditions for Figure 3

| | |
|---|---|
| Columns | 3 × Agilent PLgel 20 µm MIXED-A, 300 × 7.5 mm (p/n PL1110-6200) |
| Eluent | 1,2,4-Trichlorobenzene |
| Flow rate | 1.0 mL/min |
| Temp | 160 °C |
| Detector | RI |
| System | PL-GPC 220 |

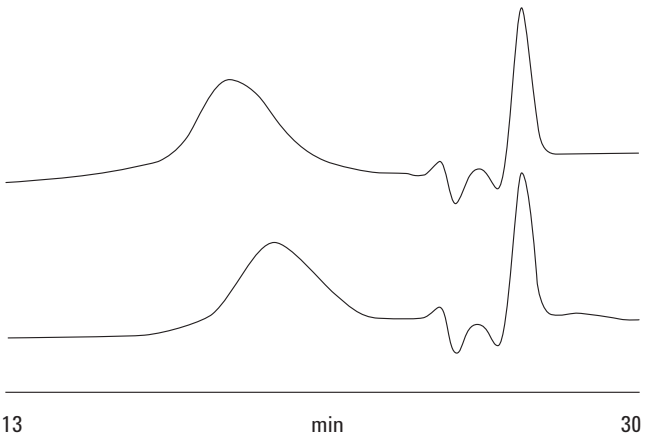


*Figure 3. Polyethylene and polypropylene separated on an Agilent PLgel 20 µm MIXED-A three-column set.*

## Conclusions

For many polyolefin-based products, solubility is limited to solvents such as trichlorobenzene at temperatures in excess of the crystalline melting point. This implies that the GPC system must be carefully temperature-controlled throughout. In these examples, temperatures between 50 and 160 °C were used. As there is no UV chromophore, RI is the most common detection technique. However, RI detection is well known for its temperature instability, and in general, dedicated integrated high temperature GPC systems are preferred. The Agilent PL-GPC 220 system is ideal for these applications. Agilent PLgel 20 µm MIXED-A columns operate successfully at the elevated temperatures required for polyolefin analysis. However, for highly crystalline polyolefins of very high molecular weight (100,000,000 g/mol polystyrene equivalent), Agilent PLgel Olexis column are preferred.

## For More Information

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